LEAD AND OTHER HEAVY METAL FIXATION IN SOILS AND SOLID WASTE BY THE MAECTITE® CHEMICAL TREATMENT PROCESS

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ABSTRACT

Lead and other heavy metals are chemically fixated using the MAECTITE® treatment process. Treatment reactions convert leachable metal species to geochemically stable and hardened mixed mineral forms within the waste matrix that are resistant to EPTOX, TCLP, MEP, acid-leach and other test methods utilized to define RCRA toxic waste and waste product longevity. Case history data from comprehensive treatability studies support process utilization on all soil types examined, debris, and solid waste consisting of battery casings, lead projectiles, sludges, slag, paint chips with abrasives, and other waste material types. Through detailed remedial work plans and other mechanisms combining engineering, geologic, soil science, and operational construction disciplines, full-scale MAECTITE® processing at RCRA, NPL, emergency response, and other lead contaminated sites on private, state, and federally funded projects have been successfully addressed. The MAECTITE® process has been patented and registered with the U.S. Patent Trademark Office.

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INTRODUCTION

Waste stabilization/solidification (S/S) methods utilize the engineered concept of physical binding mechanisms to encapsulate (micro and/or macro), entrap, absorb, contain, coat, or seal target analytes within the waste matrix. These treatment methods physically immobilize heavy metals by surface effects, plate polarity, hydration and/or adhesion principles to render waste characteristically non-hazardous (Conners, 1985)¹. To confirm treatment, S/S treated material is exposed to geotechnical test criteria that apply various physical forces or conditions to the treated mass. Failure is determined by the identification or quantification of fractures, permeability, strength, or defects. If failure occurs, physical immobilization binder mechanisms are also destroyed and new surfaces are created in the waste mass allowing for release or direct attack of formerly immobilized metal species. Further, in the presence of acidity, weakly absorbed metals may disassociate and readily diffuse to aqueous media or carrier fluid. The alleged physically immobilized mixtures generate a problem that compromise long-term stability thereby increasing inherent risk of future or current remediation costs.

While the current status of risk-free solid hazardous waste treatment for heavy metals is far from optimal efficiency (i.e., 100% removal of the total metals from the waste), many inroads to a solution have emerged. One approach is chemical treatment. With this technology, heavy metals are chemically induced to nucleate forming crystals of mineral and mixed mineral forms. These minerals are microscopic, hard, geometrically dense, acid resistant, and

will not degrade or fail under traditional geotechnical and environmental forces that prevail in landfill or natural settings. The MAECTITE® chemical process falls into the chemical treatment category.

MAECTITE® CHEMICAL PROCESS DISCUSSION

Leachable lead and other metals are chemically bound in hard and superhard mineral species that are resistant to acid leaching such as those simulated during EP toxicity leach test, toxic characteristic leaching procedure (TCLP), multiple extraction procedure (MEP) and other tests developed and approved by USEPA. Two categories (Group I and II) of treatment chemicals are usually required in variable amounts for the MAECTITE® chemical process to be effective in decreasing the TCLP lead from thousands of mg/l to less than 1 mg/l in a diverse variety of waste media.

Treatment chemicals of Group I are in the main calcium (Ca) and/or magnesium based buffers, salts and/or bases.

Treatability studies are designed and executed to identify which additive of Group I is most cost effective on a particular waste type. Treatment additives from Group I form insoluble and hard mineral species of the Barite Group in waste media. Each waste varies in chemical composition and physical characteristics that require different quantities and forms of treatment chemicals.

Treatment chemicals of Group II are capable of supplying anions that forms substantially insoluble metal compounds at normal temperature and pressure. These metal compounds preferentially nucleate and form hard and superhard, synthetic, mixed mineral species of the Apatite, Anglesite, and Pyromorphite families. Treatment chemicals of Group II can either be solid or liquid. Independent of the form, treatment chemicals of Group II preferentially convert lead (Pb) and other metals into insoluble mineral species. When constituents from both treatment chemical categories are present, thermodynamically efficient chemical reactions proceed yielding the synthetic mineral species listed in Table I. Within four (4) hours of amendment, the soluble and leachable forms of lead and other metals are chemically immobilized as hard minerals and rendered non-leachable to RCRA test methods approved by USEPA. Hard minerals have their ions tightly bonded together, thus limiting random thermal motion of the ions, resulting in increased stability and insolubility.

The combined geochemical reactions of Group I and Group II treatment chemicals with lead and other metals present in waste media lead to formation of extremely unique and mixed mineral species that are hard and compact. As a result of this and other factors, waste volume may decrease. Chemical breakdown of carbonates and bicarbonates, when present, occurs in the waste media, as a result of MAECTITE® reactions. For example, Cerrusite reacts with Group I and Group II chemicals to form extremely insoluble hydroxy lead Apatites. A decrease in waste volume as a result of the MAECTITE® chemical process has been recorded up to 55%.

The extent of decrease in leachable lead as measured by EP toxicity test and TCLP test are exhibited in Table II for different kinds of waste media. The chemical fixation of lead may exceed 99.9% when leachable lead is immobilized from 4000 ppm to 1 ppm, i.e., a decrease of less than 1/10,000 fold (0.3/4000) by both EP toxicity and TCLP test criteria.

The application of the MAECTITE® chemical treatment process for lead is universal in the sense that a wide variety of solid waste and debris such as battery casings, lead projectiles, sludges, filter cake, slag, abrasives with paint chips, plastic wire-fluff and chop, rubble, lead-dross on carbon, and all kinds of clay, soil, gravel, concrete, sand and boulders are readily treatable. Through well designed and executed treatability studies, identification and quantification of efficient Group I and Group II MAECTITE® treatment chemicals is possible.

MAECTITE® chemical process treatability studies are designed and executed with the following objectives:

- 1) verification of MAECTITE® process viability on a specific waste stream;
- 2) optimization of appropriate Group I and Group II chemicals;
- 3) determination of full-scale processing costs through engineering and pilot-scale demonstrations; and,
- 4) process control during full-scale remediation operations.

As scientific experiments, treatability studies are conducted methodically and systematically using accepted analytical and engineering principles within USEPA QA Level II data validation guidelines. Critical to successful treatability studies beyond valid analytical data generation is the replication of the MAECTITE® chemical process in a manner that simulates full-scale material handling and processing methods. Often, laboratory scaled processing

fails to encompass true site conditions, thereby miscalculating full-scale remediation and waste treatment costs.

Those practiced in the art of executing treatability studies with a background of material handling experience have traditionally generated a superior product or service. Treatability studies are summarized in final reports that provide supportable accurate quantitative, performance, cost, and design information for full-scale remediation construction onsite.

MAECTITE® treatability studies and full-scale projects completed to data have primarily focused on leachable lead. However, metals such as, barium, chromium, cadmium, selenium, nickel, and others have been successfully rendered non-leachable in a variety of waste matrices by the process. Table III presents a partial list of successfully treated metals highlighting comparative pre- and post-MAECTITE® processing analytical TCLP results.

CRYSTAL MORPHOLOGY AND STRUCTURAL STABILITY

True mineral dissolution-precipitation reactions have not been traditionally recognized as an ideal control of problem inorganic ions in soils and waste. The MAECTITE® chemical process controls problem inorganic ions through mineral dissolution-precipitation reactions and is a classic Mineralogy reaction-series approach, albeit low-temperature, that demands further emphasis as applied to RCRA toxic wastes. A large number of researchers, usually Physical Chemists, have spent enormous effort in determining and tabulating mineral solubility data and curves for pure-phase solids irrelevant to raw natural resources. Others, those unfamiliar with basic Mineralogy and Geochemistry, have taken the pure-phase solid studies as absolutes. Unfortunately, pure-phase solids are not encountered in environmental waste matrices. Non-pure and coprecipitated intertwinned solid phases are the norm (Rose et.al., 1979)². The MAECTITE chemical process is an example of this precept of nature.

Mineral solids which have analogous compositions and closely related crystalline forms, but in the same crystallographic system, are said to be isomorphous. Isomorphous minerals have various ions or molecules that enter into a greater or lesser degree the crystal-lattice of the mineral solid without causing any marked change in the crystal morphology and typically stay within the same crystallographic system. For simplicity and following Goldschmidt (1954)³, this is accomplished by the two ions having similar but not equal radii and the same charge, with the smaller ion radii being preferentially concentrated in early formed specimens of a crystallizing mineral

series. For example, a Pb ion commonly coprecipitates with a Ca ion where Pb+2 substitutes for Ca+2 within a defined crystal-lattice to form a common mineral solid (Salomons, 1975)⁴. The Pb+2 substitution for Ca+2 usually occurs based on availability of the closest ion to a vacant crystal-lattice site, in the later stages of the crystallization event when available Ca+2 has been naturally depleted, or if the available Ca+2 can be manipulated out of the system (Berner, 1971)⁵. Here, the mineralogic definition of isomorphous is very different from that of Physical Chemistry's, where isomorphous is a condition present when an ion at high dilution is incorporated by mixed (unlike) crystal formation into a precipitate, even though such formation would not be predicted on the basis of crystallographic and ionic radii. An example is precipitation of Cotunnite (Lead chloride - orthorhombic-crystallographic) with Sylvite (Potassium chloride - isometric-crystallographic)(Garrels and Christ, 1965)⁶.

In the broader sense, calcium ions are key to the MAECTITE® chemical process that forms isomorphous minerals representing the reaction-series association that exists between a series of stable mineral solids of analogous chemical formulas and crystallographic forms. Analogous formulas possess an equal number of atoms and valence charges while analogous crystallographic forms are composed of geometrically similar basic crystal-units in which an equal number of atoms are arranged in a geometrically similar manner. When the relative size of the atoms and certain physical properties (e.g. specific gravity, optical properties, etc.) are nearly the same, a potential and stable isomorphous situation exists (Burns, 1970)⁷. This concept accounts for ions or molecules, of distinctly different chemical character and even of different valence, substituting for each other within a crystal-lattice, even disordering the crystal-lattice, without typically changing the crystallographic system (Bloss, 1971)⁸.

Sometimes the crystallographic system does change. Predictably, the change is into a compatible crystallographic system prone to twinning such as the orthorhombic-crystallographic system that mimics the hexagonal-crystallographic system. This situation can be explained by Pauling's (1960)⁹ electronegativity potential. If two ions or molecules have a similar radii and the same charge, the one with the lower electronegativity potential will preferentially concentrate in early formed specimens of the crystallizing mineral suite. This explains why the orthorhombic-crystallographic Barite Group (prismatic angles of about 60° and 120° simulate the simple hexagonal-crystallographic system as a result of successive intertwinning) will crystallize from the mother-solution upon and with the hexagonal-crystallographic Apatite Group (See Figure 1). In the MAECTITE® chemical process, the

mother-solution consists of a mixture that includes waste material, leachable inorganic species, MAECTITE® reagents, and water prior to nucleation.

The MAECTITE® chemical process is an outstanding example of the formation of a low-temperature reaction-series composed of two suites of isomorphous minerals within the Apatite and Barite Groups through the manipulation of Ca+2 ions and "tag-a-long" heavy metals (See Table I). The Apatite Group represents a suite of analogous hexagonal-crystallographic compounds, primarily Apatite (Calcium phosphate), Ca-rich Pb-poor then Pb-rich Ca-poor substituted Hydroxyapatite, and Pyromorphite (Lead phosphate). The Barite Group suite of analogous orthorhombic-crystallographic compounds consists of Ca-rich Pb-poor then Pb-rich Ca-poor Anglesites and Anglesite (Lead sulfate) intertwinned. Once the sulfate ions are consumed from the mother-solution, the reaction-series shifts to a post-coprecipitation stage reverting to the Apatite Group and scavenging the remaining problematic ions to form Plumbohydroxyapatite and Pyromorphite.

All precipitation/crystallization reactions tend to carry out other constituents (ions) from the mother-solution.

Actual precipitation/crystallization occurs in a succession of steps as the MAECTITE chemical process attempts to reach equilibrium. The driving force for precipitation/crystallization is coincident crystal nucleation and heat loss.

During nucleation with expanded growth-rate, there is escalated chance of imperfections (dislocations) in the crystal's lattice-space promoting the incorporation of outside (other) ions into the crystallographic structure (Brownlow, 1979)¹⁰. The dislocation mechanism leads to the desired formation of coprecipitated isomorphous minerals in a reaction-series. As coprecipitation accelerates, the larger crystals grow at the expense of smaller crystals with the smaller crystals dissolving and reprecipitating on the surfaces of larger crystals. If the coprecipitates are not within the same crystallographic-system, the lesser mineral will form in a compatible crystallographic-system or mimic the major mineral crystallographic-system through twinning.

The process continues until equilibrium is reached with the mother-solution depleted by initial nucleation, coprecipitation and post-coprecipitation forming new distinct minerals. In effect, the problematic heavy metal ions are effectively rendered stable, insoluble and non-hazardous.

CASE HISTORY APPLICATIONS

Full-scale applications of the MAECTITE® chemical process have ranged in size from a forty-eight (48) 55-gallon drum project in one day to almost 2000 tons per day on a 150,000 ton project. Both batch and continuous processes have been used. A majority of the sites where MAECTITE® has been applied were impacted with leachable forms of lead; however, the site with the largest quantity of waste contained leachable concentrations of cadmium as the primary contaminant.

Three (3) full-scale project case histories are presented in the following discussion. The cases selected: 1) typify frequently encountered lead impacted sites found across the nation; 2) demonstrate the flexibility of the MAECTITE® chemical process on heterogeneous waste streams; and (3) create a valid data base for waste, characterized by a wide range of lead leachability (RCRA toxicity), successfully treated by the MAECTITE® chemical process.

The presented cases consist of projects in the 3000 to 15,000 ton range. Each case had lead sourced from former battery reclamation or manufacturing operations. Site waste contained toxic lead concentrations in sample extracts ranging from over 5.0 mg/l to 3659 mg/l. Oversize objects and debris were encountered within the waste matrix at each project.

Exsitu processing and treatment equipment standard for typical MAECTITE® technology applications were utilized. While early versions of the processing equipment did not apply automated controls, subsequent systems evolved to efficient, automated, low operating cost units. However, the actual material handling principles and waste-reagent mixing procedures have remained virtually unchanged.

The three (3) projects required the target waste to be prepared prior to treatment. Heavy earthmoving equipment fed staged material from a stockpile onto a barscreened hopper that was fitted with a live bottom conveyor. Debris and objects with a nominal diameter greater than four (4) inches were rejected by the screen. Material that passed through the screen into the hopper was conveyed through a dual-axled shredder for sizing and to evenly distribute the material across another conveyor belt. Material was then conveyed to a pugmill/mixer for blending with

MAECTITE® chemical reagents. MAECTITE® powder and liquid were added to the waste stream from an augered silo and the bulk liquid tank. In early equipment versions, additions of reactants were manually controlled by adjusting auger speed (powder) and delivery system valves (liquid). Evolved systems convey material from the system's shredder onto a material weigh-belt that discharges directly into an enclosed mixer.

The waste weigh-belt feeder measured waste-mass as it entered the mixer. Electronic signals from the weigh-belt were integrated by a logic controller that activated a second weigh-belt feeder for delivery of MAECTITE® powder from the silo at the prescribed dose. The controller also initiated a variable speed pump that delivered liquid MAECTITE® reagent to the mixer at the programmed flow rate. Liquid flow was monitored by a totalizing and instantaneous flow meter integrated with the logic system. A water spray was added to the mixer to help control dust and to aid mixing.

Material discharged from the mixer was transferred to staging piles located on a treatment pad where the material was covered until final disposal was executed. Prior to confirmatory treatment sampling and analysis, treated material was allowed to cure on the pad for 3-5 hours. This provided ample time for the MAECTITE® chemical process to nucleate and form root mineral crystals that chemically rendered the leachable metal non-hazardous.

Oversize material and debris were refed to the barscreen for processing or through supplemental sizing equipment as a separate step. Those final items that could not pass through the system were transferred to the site equipment decontamination pad for cleaning and manual application of MAECTITE® reagents. Items were then staged with the treated soil piles for curing and subsequent disposal as a non-hazardous waste. As a chemical treatment process, MAECTITE® complied with the RCRA BDAT rules for handling and treatment of debris.

A mobile laboratory was operated on the sites to determine if treatment was sufficient and to provide expedited sample turn-around. The onsite laboratory was also utilized to optimize waste processing variables on the various lots of material to be processed prior to there actual feeding into the system. Optimized processing minimized treatment chemical costs. The mobile laboratory adhered to a regulatory agency approved Quality Assurance Project Plan's (QAPiP) that utilized EPA QA Level II data validation procedures.

Treatment results complied with minimal USEPA treatment objectives for lead by reducing EPTOX or TCLP lead (EPA SW-846) to less than 5.0 mg/l for all projects. One case history (Site B) required compliance with EPA specified multiple extraction procedure (MEP) for lead.

SITE A

LOCATION: Indiana

Contaminant: Leachable lead (EPTOX)/lead oxide

Source of Contaminants: Battery Reclamation

Quantity of Waste: 5390 tons

Type of Waste: sand, sandy-silt, gravel, rock, and clay

Range of Total Lead: .1 to 29.9%
Range of EPTOX Lead: 20-3659 mg/L
Treatment Production: 200 tons per day

Remedial Type: Emergency Response/State Superfund

Manufacturing activity at an abandoned battery reclamation facility resulted in the emergency response action to mitigate migratory lead. Soil contaminated with lead was removed to various depths from across the site including a former neutralization soil lagoon where spent battery acids were discharged. Impacted soil containing leachable lead at concentrations up to 3659 mg/L (EPTOX) and total lead up to 29.9% from spilled lead oxide and spent battery acid was classified as a RCRA toxic waste.

Staged soils were placed in stock piles within the former facility pending waste disposal approval permitting and costing. Due to elevated off-site hazardous waste disposal costs, the MAECTITE® chemical treatment process was evaluated as a viable onsite treatment option as verified by extensive treatability studies. Soil conditions were characterized as a fairly dry (10-15% moisture) mixture of topsoil and sod, sand, silty-sand, gravel, clay, and assorted construction debris.

A treatment system mobilized to the site capable was of processing waste material at a rate of up to 500 tons per day. However, due to site space limitations and material transfer routes to curing areas, production averaged approximately 200 tons per day. Exsitu application of the MAECTITE® chemical process resulted in complete compliance with USEPA and Indiana Department of Environmental Management (IDEM) specified treatment

objectives of 5.0 mg/L EPTOX lead. Since the site was classified as an emergency response action by IDEM, a treatment permit was not required under RCRA.

The waste piles were surveyed prior to treatment and following the curing of treated material. A volume reduction of 36.4% was evidenced as a result of the MAECTITE® chemical process. The MAECTITE® chemical process appeared to create an increase in soil micelle density, destroy carbonates, dehydrate the waste matrix, and rearrange soil structure to deplete or fill voids of interstitial space.

SITE B

LOCATION: Wisconsin

Contaminant: Leachable lead (TCLP)/crushed batteries

Source of Contaminants: Battery Reclamation

Quantity of Waste: 11,000 tons

Type of Waste: silty-sand, gravel, clay, rock, casing ash

Range of Total Lead: 2.03 to 3.55% Range of TCLP Lead: >5.0-422.4 mg/L Treatment Production: 400 tons per day

Remedial Type: Emergency Response/Federal Superfund

Approximately 11,000 tons of soil were excavated from within an old quarry location that held a former battery cracking and lead reclamation operation. Soils were contaminated with leachable lead from the mishandling of spent battery acids, battery casings, and casing combustion byproducts. Total lead concentrations ranged up to 3.55%. TCLP lead exceeded the RCRA limit and ranged as high as 422.4 mg/L.

A treatability study was executed on hazardous waste that exceeded the initial RCRA test method of 5.0 mg/L via EPTOX procedures and EPA's proposed treatment objective of 0.51 mg/L via TCLP methodology. At the completion of the study, EPA adopted the treatment objective of 5.0 mg/l by TCLP as the final lead treatment performance criteria nation-wide. The treatability study was revisited to optimize the process for the final treatment standard. Results demonstrated that the waste was readily treated to <5.0 mg/L TCLP lead. A volume reduction of 23.4% was predicted for full-scale processing. This was important in designing the onsite special waste cell where treated material was to be interned.

Based upon the treatability study results, and a production capacity of 500 tons per day, a MAECTITE® treatment system was mobilized to the site, setup and optimized. In addition to onsite treatment, treated material was placed in a clay lined cell constructed onsite in accordance with a design approved by both the USEPA and the Wisconsin Department of Natural Resources.

Treatment operations commenced in the fall (October) and were completed in January. Due to extreme cold weather (<-15°F) the treatment production averaged only 400 tons per day. Although cold weather created adverse working conditions for site workers and drastically impaired the operation of diesel powered equipment, MAECTITE® reactions were extended over a 5-7 day period instead of the typical 3-5 hours. The severe cold turned the MAECTITE® liquid reagent into a gel hindering accurate volumetric dosing and subsequent thorough mixing with the waste. This was addressed by corrective operation actions.

A volume reduction of 22.4% was determined by certified site surveys conducted on the pretreated pile and the post-treated placed material. Since material was compacted with a sheepsfoot after placement in the cell, the 22.4% reduction was calculated after accounting for a 30% reduction due to mechanical compaction.

SITE C

LOCATION: South Dakota

Contaminant: Leachable lead (TCLP)/crushed batteries

Source of Contaminants: Battery Reclamation

Quantity of Waste: 4,000 tons

Type of Waste: sod, silty-sand, loess, clay

Range of Total Lead: up to 2.0%
Range of TCLP Lead: >5.0-85 mg/L
Treatment Production: 500 tons per day

Remedial Type: Federal Superfund/PQOPS-FSS

Approximately 2200 cubic yards of soil and battery casings were excavated and staged in a pile onsite by a USEPA ERCS contractor. The staged material was sealed with a foam to prevent dust migration. The site was under the first PreQualified Offerors Procurement Strategy (PQOPS) program contract issued by the USEPA in Washington, DC. The contract specified the treatment and removal of the stockpiled material in addition to underlying soils. In total, approximately 4000 tons of material were treated with the MAECTITE® chemical process and disposed at the local municipal non-hazardous landfill facility.

All material treated onsite was rendered non-hazardous with all TCLP lead results <0.1 mg/L and a cumulative MEP (SW-846, Method 1320) concentration (sum of all 10 leaches) less than 1.0 mg/L in each sample. USEPA specified the MEP test in order to determine if treated material would withstand simulated exposure to 1000 years of acid rain or acidic leachate within non-hazardous landfill cells. USEPA also specified that no single extraction of an MEP test could contain more than 5.0 mg/L lead versus the more stringent results that were actually obtained with the MAECTITE® chemical process (See Table IV).

Treated material was transported to the local licensed municipal landfill as a non-hazardous waste. No material disposed offsite was classified hazardous waste under the "Debris Rule" exemption. Oversize objects were either sized by the processing equipment or manually treated on the equipment decontamination pad, and then placed in the staged piles of treated material for disposal as a non-hazardous waste.

SUMMARY

The MAECTITE® chemical process creates a low-temperature reaction-series of stable isomorphous insoluble mineral species. Waste containing leachable lead and other heavy metal ions are rendered non-hazardous by RCRA definition. The end treatment product contains hard and super hard minerals that are resistant to acidity, and degradation by geotechnical forces and the chemical conditions associated with landfill and natural settings. Processing of waste often results in a volume reduction. Successful full-scale projects have been completed for private, state and Federal interests. As a demonstrated state-of-the-art chemical process, soils and solid waste containing leachable heavy metals may be easily treated using insitu or exsitu processing equipment. To date, no lead containing waste has been found resistant to MAECTITE® chemical treatment.

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TABLE I

Synthetic Mineral Species of Lead Detected in a Treated Sample (Listed in Decreasing Order of Abundance)

Abundance	Crystal System	Isomorphic Groups	Mineral Species
31-41%	Hexagonal	(A)	Calcium Substituted Hydroxy Lead <u>Apatite</u> , Ca _{0.5-1.5} Pb _{3.5-4.5} (OH)(PO ₄) ₃
28-29%	Hexagonal + Orthorhombic	(A) + (B)	Mixed Calcium Lead Phosphate Sulfates, Ca _{0.05-0.2} Pb _{0.8-0.95} (PO ₄) _{0.15-0.5} (SO ₄) _{0.25-0.75}
21-22%	Orthorhombic	(B)	Calcium Anglesite, Ca _{0.05-0.3} Pb _{0.7-0.95} SO ₄
3-6%	Orthorhombic	(B)	Anglesite, PbSO ₄
2-7%	Hexagonal	(A)	Lead Hydroxy/Chlor Apatite, Pb ₅ (PO ₄) ₃ (OH) _{0.5} Cl _{0.5}
1-3%	Hexagonal	(A)	Pyromorphite, Pb ₃ (PO ₄) ₂
1-2%	Hexagonal ^R	(A)*	Organo-Lead Phosphate Sulfate, (Unnamed) Humus-o-Pb ₃ (PO ₄)(SO ₄)

Apatite Group (A) (B) Barite Group

Complexation by Organic is dictated by the Crystal System, a Pseudo-rhombohedral of the Hexagonal System

Pseudo-Rhombohedral of the Hexagonal System R

Mineralogic assay conducted by x-ray fluorescence spectrometry, polarized light microscopy (PLM) and scanning electron Note

microscopy (SEM) methods on battery reclamation site soils and waste after one year from treatment by the MAECTITE® chemical

process

TABLE II APPLICATION OF MAECTITE® CHEMICAL TREATMENT PROCESS TO A DIVERSE VARIETY OF WASTE MEDIA

WASTE TYPE	TOTAL	LEACHABLE LEAD (mg/l)		
(MATRIX)	LEAD %	Before Treatment	After Treatment	
Sandy loam	2.2	163.7	1.5	
Lead birdshot	16.1	3,720	ND	
Lead buckshot	11.4	1,705	ND	
Clayey slag	14.6	91.8	ND	
Slag-lead smelter	6.6	21.3	2.0	
Topsoil	15.8 14.6 0.344	44.5 91.8 83.5	1.4 ND 0.5	
Silty sand/debris	0.56	34.6	ND	
Battery casings	0.6 - 12 2.0	288 160	0.6 0.3	
Organic humus soil	0.31 - 1.9	23.2	ND	
Silty sand	4 - 5	687	0.7	
Solid waste	1.1 0.4	9.7 72.4	0.01 3.4	
Sludge-industrial waste	2.2	59.3	1.6	
Filter cake	2.9	245.3	1.1	
Gravel	0.16	7.5	0.5	
Road gravel	0.34	46	ND	
Gray clay	2.2	495	0.2	
Grayish brown ash	9.5	520	0.3	
Brown soil-gravel clay (till) Brown soil-gravel sand (till)	1.37 3.97	263 303	2.1 1.6	
Soil with PbO	29.9	3,659	ND	
Clarifier sludge	0.85	57.1	0.3	
RCRA organic sludge	9.4	580	ND	
Carbon with lead dross	12.6	105.6	0.5	
Foundry sand with bentonite	1.96	461.2	ND	
Wire fluff	0.33 - 0.134	15.9 - 130	0.7	
Wire chip	0.3 - 0.7	28	1.9	

NOTE: ND = Not Detected (i.e., <0.5 mg/l)

BDL = Below Detection Limit (i.e. <0.1 mg/l)

Listed results from bench-, engineering-, and full-scale application

All analytical procedures performed in accordance with SW-846 (USEPA)

TABLE III

CHEMICAL TREATMENT OF VARIOUS IONS BY MAECTITE® PROCESS

Metal Type	Application (level)	Untreated Conc. Range	Treated Conc. Range	Regulatory Limit
Lead	Commercial (full-scale)	> 5 to 3720	< 1	5
Cadmium	Commercial (full-scale)	> 1 to 1596	< 0.1 to 0.7	1
Selenium	Commercial (bench-scale)	> 1 to 300	<0.0025	1
Chromium*	Commercial (full-scale)	> 5 to 660	< 0.3	5
Nickel	Laboratory (bench-scale)	> 5 to 250	< 0.5	N.R.
Barium	Laboratory (bench-scale)	> 400	< 10	100

N.R. = Not Regulated under RCRA "Land Ban" criteria

- * Chromium (VI) was treated by the patent-pending, proprietary Chromtite TM treatment technology that is applicable to multivalent species. Chromtite TM is a sister technology to MAECTITE®.
- All analytical units in mg/l in TCLP extract

TABLE IV

MULTIPLE EXTRACTION PROCEDURE TEST RESULTS FOR LEAD ON MAECTITE® TREATED WASTE MATERIAL AT SITE C UNDER THE PQOPS PROGRAM

Sample Number	Mobile Lab*	EPA CLP Lab	
CTB 1 & 2	ND (0.88 mg/l)	.850 mg/l	
CTBP 3 & 4	ND (0.56 mg/l)		
CTBP 3 & 4 (DUP)	ND (0.37 mg/l)		
CTBP 5 & 6	0.61 mg/l		
CTBP 7 & 8	0.54 mg/l		
CTBP 9 & 10	0.78 mg/l		
CTBP 11 & 12	0.83 mg/l		
CTBP 13 & 14	ND		
CTBP 15 & 16	0.51 mg/l		

* Mobile Laboratory

Method: MEP Test (EPA Method 1320)

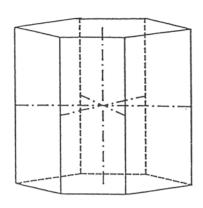
Detection Limit: 0.50 mg/l

** EPA CLP Lab

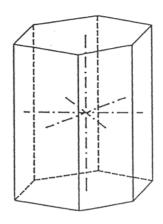
Analytical completed for 10% of samples Analysis for confirmation purposes only Method: MEP Test (EPA Method 1320)

Detection Limit: 0.025 mg/l

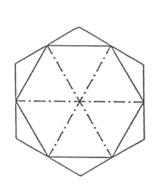
FIGURE 1 CRYSTALLOGRAPHIC TWINNING MAECTITE® CHEMICAL PROCESS



HEXAGONAL SYSTEM FIRST ORDER PRISM



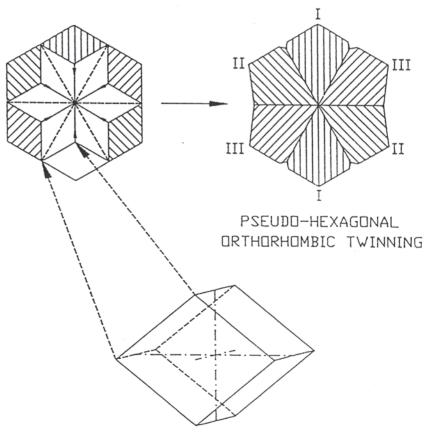
HEXAGONAL SYSTEM SECOND ORDER PRISM



HEXAGONAL SYSTEM (Top View)



RHOMBOHEDRAL DIVISION OF HEXAGONAL SYSTEM



ORTHORHOMBIC SYSTEM